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**09/647207** 430 Rec'd PCT/PTO 27 SEP **2000** 

**HUBR 1165** 

# IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Applicant(s)

Stuke et al.

International

Appln. No.

PCT/EP99/02238

Filed

Herewith

For

ADHESIVE-FREE POLYMER COMPONENT JOINTS FOR

PRODUCING CLOSED MICRO- AND NANO-CHANNEL

**STRUCTURES** 

Hon. Commissioner of Patents

September 26, 2000

and Trademarks

Washington, D.C. 20231

# PRELIMINARY AMENDMENT

SIR:

Prior to examination, please amend the application as follows:

# IN THE CLAIMS

Please cancel pending claims 1-22.

Please add the following claims 23-44, as follows:

- 23. A process for the adhesive-free production of polymeric components, including the steps of:
  - (a) preparing a polymeric substrate which, on at least one surface, has depressions forming micro- and/or nanochannel structures,

- (b) applying, by uniform pressure extending over the surface, a polymeric covering to a surface present on the substrate, said substrate having depressions but otherwise being smooth,
- (c) slowly heating the substrate, with the covering applied by pressure, to a temperature which is at least as high as the glass transition temperature of the substrate and/or of the covering, for the bonding thereof, and
- (d) cooling.
- 24. The process as claimed in claim 23, wherein the polymeric substrate and the polymeric covering are selected from the group consisting of acrylic polymers, polycarbonates, polystyrenes, and also copolymers and mixtures of these.
- 25. The process as claimed in claim 24, wherein the polymeric substrate and the polymeric covering are selected from the group consisting of acrylic polymers, in particular of polymethyl methacrylate polymers, or of polymeric carbonates.
- 26. The process as claimed in claim 23, wherein the substrate has depressions with a width or/and depth within the range from 10 nm to 2 mm.

27. The process as claimed in claim 26, wherein

the substrate has depressions with a width or/and depth within the range from  $100\,\mathrm{nm}$  to  $1\,\mathrm{mm}$ .

- 28. The process as claimed in claim 27, wherein the substrate has depressions with a width or/and depth within the range from 1  $\mu m$  to 500  $\mu m$ .
- 29. The process as claimed in claim 23, wherein substrate and covering are selected from among polymeric materials of the same type.
- 30. The process as claimed in claim 23, wherein at least the covering is selected from among optically transparent materials.
- 31. The process as claimed in claim 23, wherein the polymeric covering and the substrate are combined by pressure.
- 32. The process as claimed in claim 31, wherein the pressure applied is within the range from 1 to  $1000 \text{ kg/cm}^2$ .
- 33. The process as claimed in claim 23, wherein the duration of heating is within the range from 0.5 to 3 h.
- 34. The process as claimed in claim 23, wherein the heating temperature is not more than 5°C above the glass transition temperature.
- 35. The process as claimed in claim 23, wherein the substrate and the covering present thereupon are held within the region of the heating temperature for a period of at least 15 min.

- 36. The process as claimed in claim 35, wherein the substrate and covering present thereupon are held within the region of the heating temperature for a period of at least 30 min.
- 37. The process as claimed in claim 35, wherein the holding temperature is within  $\pm 3$  °C of the heating temperature.
- 38. The process is claimed in claim 23, wherein the duration of the cooling is at least 1 h.
- 39. The process as claimed in claim 38, wherein the duration of the cooling is at least 2 h.
- 40. The process as claimed in claim 23, wherein the duration of the cooling is up to 30 sec.
- 41. A polymeric constituent with hollow structures present therein, obtainable by a process as claimed in claim 23.
- 42. A polymeric component as claimed in claim 41, wherein the hollow structures comprise closed channels with a width or/and depth of from 10 nm to 10 mm.
- 43. A polymeric component as claimed in claim 41, wherein the interior of the component is free from adhesives.
- 44. The use of polymeric components as claimed in claim 41 in detection procedures, in particular in optical or/and electrical detection procedures.

# **REMARKS**

This application is a national stage filing under 35 U.S.C. § 371 of PCT/EP99/02238. Support for new claims 23-44 is found in the English-language translation of the specification, in the English-language translation of originally filed claims 1-22, and in the translation of the annex to the International Preliminary Examination Report. Applicants request entry of the foregoing amendments.

A check in the amount of \$507.00 is included with this filing to cover the basic national fee, and the fee for two additional claims over 20 claims. Fees have been reduced by half, reflecting applicant's small entity statement. A small entity statement is filed herewith. The Commissioner is hereby authorized to deduct any additional fees associated with this filing from, or credit any overpayment to Deposit Account No. 500624.

Respectfully submitted,

FULBRIGHT & JAWORSKI L.L.P.

David Rubin

Reg. No. 40,314

666 Fifth Avenue New York, New York 10103 (212) 318-3000

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Adhesive-free bonding of polymeric components to produce closed micro- and nanochannel structures

## Description

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The invention relates to a process for producing polymeric components with hollow structures present therein, e.g. in the form of closed micro- or/and nanochannels, said process using no adhesives. The invention further relates to the polymeric parts obtainable by the process and to their use in detection procedures.

Polymeric components, e.g. plastic biochips, interior of which closed hollow structures are present have hitherto been produced by a process in which an adhesive, e.g. a UV-curable adhesive, has been used to bond a plastic outer layer onto a plastic substrate in which depressions are present. However, the use of the adhesive has led to considerable disadvantages. For example, if too much adhesive was applied, capillary interactions caused it to migrate into the channels and render these impassable, at least to some extent. On the other hand, if too little adhesive was used dead spaces were produced directly adjacent to the channels. The process was moreover highly inconvenient, since operations had to be carried out under a microscope. Finally, the presence of the adhesive also impaired the chemical or/and spectroscopic properties of the plastic component.

DE-A-40 22 793 has disclosed that a heated welding tool can be used to weld a polymeric film onto a sheet of polymer in which recesses are present, without prior heating of the sheet of polymer or the polymeric film. The pressure of the welding tool produces a grid of point welds. The welding tool is heated to a temperature of from 250 to 300°C (column 4, lines 63-65), and therefore chemical modification of the

polymeric materials can occur, combined with possible reduction in transparency and/or increase in base-level fluorescence. In addition, undesirable dead spaces are produced adjacent to the welds.

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The object on which the present invention is based was therefore to provide a process for producing polymeric or plastic components provided with hollow structures, said process at least to some extent avoiding the abovementioned disadvantages of the prior art.

This object is achieved by a process for producing polymeric constituents, including the steps of:

- (a) preparing a polymeric substrate which has depressions on at least one surface,
- (b) applying a polymeric covering to a surface present on the substrate and having depressions,
- (c) heating the substrate with the covering present thereupon to a temperature which is at least as high as the glass transition temperature of the substrate or/and of the covering, and
- (d) cooling.

Step (a) of the novel process comprises preparing a polymeric substrate with open depressions on a surface. 25 A covering is applied to this surface with the aim of producing a polymeric component with hollow structures closed on their upward-facing sides. The polymeric substrates and polymeric coverings used for this 30 purpose are selected from the class consisting of meltprocessable thermoplastics, preferably from the class consisting of acrylic polymers, polycarbonates, polystyrenes, and also copolymers and mixtures of these. It is preferably for polymeric substrates and polymeric covering to be selected from among acrylic 35 polymers, such as polyacrylate, polymethacrylate and in particular polymethyl methacrylate polymers polycarbonates.

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The polymeric substrate has depressions on at least one surface. These depressions preferably have a width or/and depth within the range from 10 nm to 2 mm, particularly preferably from 100 nm to 1 mm and most preferably from 1  $\mu$ m to 500  $\mu$ m. The depressions preferably comprise structures in the form of channels.

Using the processaccording to the invention, a polymeric covering, for example a polymeric film, is laminated onto this substrate without using adhesives. For this, substrate and covering are preferably selected from among polymeric materials of similar type, in particular from the same polymeric materials. It is moreover preferable for at least the covering and in particular both the covering and the substrate to be composed of optically transparent materials, i.e. materials transparent within the visible or/and UV light regions.

20 produce the substrate with a surface having depressions, a contact mask may first be produced, namely by using a laser to etch the desired microstructures into a silicon membrane under chlorine gas. This contact mask is then laid on the plastic substrate and irradiated with laser light, e.g. with a 25 UV vacuum laser, whereupon ablation cuts the desired channels into the plastic. The depth of cut may be set precisely via the laser and is, for example, 100 nm per irradiation. The resultant channels have a very smooth 30 surface. Removal of the mask then gives the polymeric substrate which can be used for the process according to the invention. As an alternative, the substrates provided with open microstructures may also be produced from a master mold, e.g. by injection molding. 35

Step (b) of the process according to the invention comprises the application of a polymeric covering onto one or more surfaces present on the substrate and having depressions. For this, the surface of the

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polymeric covering, which may also be a film, for example, and the surface of the substrate are prepared in a form which is clean and as smooth as possible. The covering is then preferably positioned on the substrate and the two parts combined by pressure, the pressure applied being preferably within the range from 0.1 to  $1000 \text{ kg/cm}^2$ , e.g. from 0.2 to  $20 \text{ kg/cm}^2$ .

in step (c) of the process according to the invention, the substrate, with the covering present thereupon, is heated to a temperature which is at least as high as the glass transition temperature of the of substrate or/and the covering. The heating preferably takes place in a controllable heating cabinet, proceeding slowly from the initial temperature (e.g. room temperature) to a value just above the glass transition temperature of one of the polymers. glass transition temperature depends on the heating rate and can readily be determined for various materials by the skilled worker by simple experimentation. The duration of heating is preferably within the range from 0.5 to 3 h, particularly preferably within the range from 0.5 to 1.5 h. heating temperature is preferably within a range between the transition temperature glass and temperature which is 5°C above the glass transition temperature. The heating temperature is particularly preferably within a range between 0.5 and 3°C above the glass transition temperature.

Once the heating temperature has been reached, substrate and the covering present thereupon preferably held for a particular period within

preferably held for a particular period within the range of the heating temperature. This period is preferably at least 15 min, particularly preferably at least 30 min, for example from 40 to 45 min. The holding temperature is preferably within approximately ±3°C of the heating temperature.

Step (d) of the novel process comprises the cooling. The cooling to about 40°C is preferably carried out slowly. The duration of the cooling is generally at least 1 h, particularly preferably at least 2 h and most preferably up to 3.5 h. As an alternative, the cooling may also take place within a few seconds, e.g. up to 30 sec. After the cooling, the finished polymeric part can be removed.

process gives adhesive-free bonding novel form of polymeric coverings, preferably in the structured, preferably films, and transparent polymeric substrate. transparent, sheets of bonding is mechanically and chemically stable. carried out at relatively low can be process temperatures in the vicinity of the glass transition temperature, preferably just above the glass transition temperature. No reaction products are produced, and the process is therefore extremely clean and biocompatible: reduced measurements show no particular, increased fluorescence and no in transparency resultant component. If the covering materials and substrate materials are of the same type, the component produced is composed of just a single material and has optical and electrical properties superior to those of multicomponent systems. The optical quality is so high that it is even possible to detect individual molecules in channels of the components with a good signal/noise ratio.

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The present invention further provides a polymeric component with hollow structures present therein, the component being obtainable by the process described above. The hollow structures present in this polymeric component are preferably closed channels, i.e. channels closed on their upward-facing sides, with a width or/and depth of from 10 nm to 2 mm, and the component differs from polymeric parts known from the prior art in that it is essentially or even completely free from

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adhesives and from thermal reaction products in its interior, in particular in the region of the hollow structures. The novel polymeric part also has full-surface bonding in the region where the surfaces of substrate and covering are in contact, so that no dead spaces are present in the region of the hollow structures. The novel polymeric part may be used for detection procedures, in particular in optical or/and electrical detection procedures.

The invention is further described by the examples below.

# Example 1 Production of a polymethyl methacrylate component

A PMMA film is positioned on a surface of a PMMA substrate block, which surface has micronanochannel structures. The surfaces of both parts are clean and smooth. The two parts are placed between two flat sheets of glass, which are then clamped into a press. The pressure applied in the press is within the range from 0.2 to 20 kg/cm<sup>2</sup>, e.g. 2 kg/cm<sup>2</sup>. The entire system is then slowly heated, preferably within a heating time of from 0.5 to 1.5 h, in a controllable heating cabinet, to just above the glass transition The glass transition temperature of the polymer. temperature here depends on the heating rate. The ideal bonding temperature for the heating rate mentioned is  $106 \pm 0.5$ °C.

The system is then held for a period of from 40 to 45 min at a temperature between  $104\,^{\circ}\text{C}$  and the ideal bonding temperature. This is followed by slow cooling, preferably for  $\leq 3.5$  h. After the cooling, the finished structure can be removed from the apparatus. If desired, the cooling phase may also be considerably shortened, down to the seconds region.

# Example 2 Production of a polycarbonate component

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Using the method described in Example 1, a polycarbonate component was produced. It was found here that this material, too, was suitable for producing components with closed micro- and nanochannel structures.

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The bonding temperature was within the range from 150 to 160°C.

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## Claims

- 1. A process for producing polymeric constituents, including the steps of:
- (a) preparing a polymeric substrate which has depressions on at least one surface,
  - (b) applying a polymeric covering to a surface present on the substrate and having depressions,
- 10 (c) heating the substrate with the covering present thereupon to a temperature which is at least as high as the glass transition temperature of the substrate or/and of the covering, and
- 15 (d) cooling.
  - 2. The process as claimed in claim 1, wherein

the polymeric substrate and the polymeric covering are selected from the group consisting of acrylic polymers, polycarbonates, polystyrenes, and also copolymers and mixtures of these.

3. The process as claimed in claim 2,

25 wherein

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the polymeric substrate and the polymeric covering are selected from the group consisting of acrylic polymers, in particular of polymethyl methacrylate polymers, or of polymeric carbonates.

4. The process as claimed in any of claims 1 to 3, wherein

the substrate has depressions with a width or/and depth within the range from 10 nm to 2 mm.

5. The process as claimed in claim 4, wherein

the substrate has depressions with a width or/and depth within the range from 100 nm to 1 mm.

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6. The process as claimed in claim 5,

# wherein

the substrate has depressions with a width or/and depth within the range from 1  $\mu m$  to 500  $\mu m\,.$ 

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7. The process as claimed in any of the preceding claims,

#### wherein

- substrate and covering are selected from among polymeric materials of the same type.
  - 8. The process as claimed in any of the preceding claims,
- 15 wherein

at least the covering is selected from among optically transparent materials.

9. The process as claimed in any of the preceding claims,

#### wherein

the polymeric covering and the substrate are combined by pressure.

25 10. The process as claimed in claim 9,

## wherein

the pressure applied is within the range from 1 to 1000  $\rm kg/cm^2\,.$ 

30 11. The process as claimed in any of the preceding claims,

# wherein

the duration of heating is within the range from 0.5 to  $3~\mathrm{h}.$ 

12. The process as claimed in any of the preceding claims,

wherein

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the heating temperature is not more than 5°C above the glass transition temperature.

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The process as claimed in any of the preceding 13. 5 claims,

#### wherein

the substrate and the covering present thereupon the heating are held within the region of temperature for a period of at least 15 min.

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The process as claimed in claim 13, 14. wherein

> the substrate and the covering present thereupon are held within the region of the heating temperature for a period of at least 30 min.

- The process as claimed in claim 13 or 14, 15. wherein
- the holding temperature is within ±3°C of the heating temperature. 20
  - The process as claimed in any of the preceding 16. claims,

#### wherein

- the duration of the cooling is at least 1 h. 25
  - 17. The process as claimed in claims 16, wherein

the duration of the cooling is at least 2 h.

- 18. The process as claimed in any of claims 1 to 15, wherein the duration of the cooling is up to 30 sec.
- A polymeric constituent with hollow structures 35 present therein, obtainable by a process claimed in any of claims 1 to 18.
  - A polymeric component as claimed in claim 19,

# wherein

the hollow structures comprise closed channels with a width or/and depth of from 10 nm to 10 mm.

5 21. A polymeric component as claimed in claim 19 or 20,

### wherein

the interior of the component is free from adhesives.

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22. The use of polymeric components as claimed in any of claims 19 to 21 in detection procedures, in particular in optical or/and electrical detection procedures.

# - 12 -Abstract

The invention relates to a process for producing polymeric components with hollow structures present 5 therein, e.g. in the form of closed micro- or/and nanochannels, said process involving no use of adhesives. The invention further relates to the polymeric parts obtainable by the process and the use of these in detection procedures.

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Additional inventors are being named on the

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